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Date

PCT/EP2005/

PAI.51128/WO

00 44 1753 877123

11 October 2005

003047

**Dear Sirs** 

Re. International Application PCT/EP2005/003047

Title: Coating Compositions based on Modified Epoxy Resins

In the name of Imperial Chemical Industries

Our ref: PAI 51128/WO

Applicant wishes to make the following comments and amendments under Art.34 (PCT) relating to the Written Opinion of the International Searching authority.

## **Application in Suit**

The application in suit addresses the problem of reducing the amount of extractable bis phenol A diglycidyl ether in crosslinked coatings. This is achieved by forming

- a protective crosslinkable coating composition comprising modified epoxy resin and crosslinker the modified epoxy resin being the reaction product, by weight, of
- i) from 80 to 99.9 parts of di-epoxy resin of epoxy equivalent weight from 500 to 5000 and formed from the reaction of bis phenol A diglycidyl ether and bis phenol A and
- ii) from 0.1 to 20 parts of reactive material

characterised in that

a) the di-epoxy resin contains minor amounts of resin components of molecular weight less than 1000 Daltons

and the reactive material comprises

- b) mono-functional organic material of molecular weight at least 100 Daltons having one moiety capable of reacting with the epoxy moieties of the di-epoxy resin and
- c) dicarboxylic acid of molecular weight less than 300 Daltons having two moieties capable of reacting with the epoxy moieties of the di-epoxy resin

## D1 (US 4686248)

D1 discloses a water thinnable binder which is the reaction product of a multifunctional polyglycidyl ether and a dicarboxylic acid, having an acid value of at least 20mg KOH/g. The polyglycidyl ether has n epoxy groups (where 1≤n<2).

There is no disclosure in D1 that the polyglycidyl ether should be reacted with a monofunctional organic material in addition to the dicarboxylic acid. This is required in the application in suit as it enables the BADGE level to be significantly reduced whilst ensuring that the viscosity of the modified diepoxy resin does not rise which would limit the solids of the coating (page 8, line 24-26). All the examples in D1 react the polyglycidyl ether with only dicarboxylic acid.

Furthermore, the application in suit requires that the diepoxy resin is formed from the reaction of bis phenol A diglycidyl ether and bis phenol A. It is evident from the description and the claims that the diepoxy resins used in D1 are not derived from BADGE and thus the teaching of D1 cannot have anything to do with reducing extractable BADGE from can coatings. Thus applicant's claim 1 is novel and inventive over D1.

# **D2 (US 4098735)**

D2 discloses the reaction product of a polyglycidyl ether, and a mono and dicarboxylic acid. The mono-acids have between 8-18 carbon atoms. The dicarboxylic acid-has from 4 to 8 methylene groups. The ratio of monocarboxylic acid:dicarboxylic acid is from 1:2 to 1:5 (based on equivalent.weight). On a molar basis this is 1:4 to 1:10.

The monocarboxylic acid of D1 is equivalent to the monofunctional organic material of the application in suit and the dicarboxylic acid in each description are equivalent.

So whilst existing claim 1 is not novel over the disclosure of D2, existing claim 2 of the application in suit is novel over D2 as the ratio of monofunctional organic material to dicarboxylic acid of 3:1 to 12:1 is not disclosed in D2. The importance of this ratio is to achieve the optimum balance between the solids content of the liquid coating and adverse flavour effects on the can contents as explained at page 8, line 21-29. Thus claim 2 is also inventive.

Existing claim 7 as dependent from claim 1, is novel too, because D2 does not disclose tartaric acid, which has two methylene groups. As indicated above, D2 discloses only dicarboxylic acids of from 4 to 8 methylene groups.

# New Claims and Basis

Thus existing claims 1-15 are deleted and a new set of claims 1-15 is submitted (with a set in manuscript form for the examiner's convenience).

Basis for the new claims can be found in the application as filed as indicated below.

New claim	Basis
1	Existing claim 1 plus added limiting feature found at page 8, line 28
2	Existing claim 1 plus added limiting feature found at page 8, line 2
3	Page 5, line 18
4	Page 5, line 29 to page 6, line 1
5	Page 7, line 16-17
6	Page 8, line 12-13
7	Page 8, line 1-2
8	Page 7, line 5
9	Page 6, line 24-26
10	Page 10, line 11-16
11	Page 10, line 20-21
12	Page 10, line 8-9
13	Whole document
14	Whole document

The description is also amended according to the new claims and replacement pages 5 and 5a are provided in manuscript and typed form for the convenience of the examiner.

The international preliminary examination should proceed based on the new claims.

Yours faithfully

Dr A Frangou

Legal Affairs Department

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#### Claims

- 1) A protective crosslinkable coating composition comprising modified epoxy resin and crosslinker the modified epoxy resin being the reaction product, by weight, of
- i) from 80 to 99.9 parts of di-epoxy resin of epoxy equivalent weight from 500 to 5000 and formed from the reaction of bis phenol A diglycidyl ether and bis phenol A and
- ii) from 0.1 to 20 parts of reactive material

# characterised in that

a) the di-epoxy resin contains minor amounts of resin components of molecular weight less than 1000 Daltons

and the reactive material comprises

- b) mono-functional organic material of molecular weight at least 100 Daltons having one moiety capable of reacting with the epoxy moieties of the di-epoxy resin and
- c) dicarboxylic acid of molecular weight less than 300 Daltons having two moieties capable of reacting with the epoxy moieties of the di-epoxy resin and where the ratio of mono-functional organic material to dicarboxylic acid calculated on a molar basis is from 3:1 to 12:1.
- 2) A protective crosslinkable coating composition comprising modified epoxy resin and crosslinker the modified epoxy resin being the reaction product, by weight, of
  - from 80 to 99.9 parts of di-epoxy resin of epoxy equivalent weight from 500 to 5000 and formed from the reaction of bis phenol A diglycidyl ether and bis phenol A and
  - ii) from 0.1 to 20 parts of reactive material

## characterised in that

a) the di-epoxy resin contains minor amounts of resin components of molecular weight less than 1000 Daltons

and the reactive material comprises

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- b) mono-functional organic material of molecular weight at least 100 Daltons having one moiety capable of reacting with the epoxy moieties of the di-epoxy resin and
- c) tartaric acid having two moieties capable of reacting with the epoxy moieties of the di-epoxy resin
- 3) A coating composition according to claim 1 or 2 characterised in that the resin component of molecular weight less than 1000 Daltons comprises bis phenol A diglycidyl ether.
- 4) A coating composition according to claim 3 characterised in that the amount of bis phenol A diglycidyl ether extractable from a crosslinked coating of the coating composition is less than 0.3 micrograms/dm<sup>2</sup>
- 5) A coating composition according to any one of the preceding claims characterised in that the mono-functional organic material is a mono-carboxylic acid.
- 6) A coating composition according to claim 5 characterised in that the mono-carboxylic acid is tetradecanoic acid.
- 7) A coating composition according to any one of claims 1, 3 to 6 characterised in that the di-carboxylic acid is tartaric acid.
- 8) A coating composition according to any one of the preceding claims characterised in that the amount of reactive material comprises from 1 to 20% by weight of the modified epoxy resin.
- 9) A coating composition according to any one of the preceding claims characterised in that the modified epoxy resin has at least 30% of the number of epoxy groups as on the diepoxy resin from which it is derived.
- 10) A process for producing the modified epoxy resin as defined in any one of the preceding claims comprising the steps of causing a diepoxy resin of epoxy equivalent weight of from 500 to 5000, formed by the reaction of bis phenol A diglycidyl ether and bis phenol A and containing minor amounts of resin components of molecular weight less than 1000 Daltons to react with a mono-functional organic material of molecular weight at least 100 Daltons and a dicarboxylic acid of molecular weight less than 300 Daltons.
- 11) A process according to claim 10 characterised in that the mono-functional organic

material is reacted with the diepoxy resin in a first step, the resulting product being reacted with the dicarboxylic acid in a later step.

- 12) A metal container coated with the coating composition according to any one of claims 1 to 9.
- 13) A process of producing a crosslinked coating on a metal container characterised in that it comprises the steps of applying a coating according to any one of claims 1 to 9 and causing the coating to crosslink.
- 14) A modified epoxy resin as defined in any one of claims 1 to 9.
- 15) The use of a modified epoxy resin for reducing the amount of bis phenol A diglycidyl ether extractable from a crosslinked coating composition on the interior surface of a metal container to less than 0.3 micrograms/dm<sup>2</sup>, said modified epoxy resin being as defined in claim 14.

Claims

- 1) A protective crosslinkable coating composition comprising modified epoxy resin and crosslinker the modified epoxy resin being the reaction product, by weight, of
- i) from 80 to 99.9 parts of di-epoxy resin of epoxy equivalent weight from 500 to 5000 and formed from the reaction of bis phenol A diglycidyl ether and bis phenol A and
  - ii) from 0.1 to 20 parts of reactive material

characterised in that

a) the di-epoxy resin contains minor amounts of resin components of molecular weight less
than 1000 Daltons

and the reactive material comprises

- b) mono-functional organic material of molecular weight at least 100 Daltons having one moiety capable of reacting with the epoxy moieties of the di-epoxy resin and
- c) dicarboxylic acid of molecular weight less than 300 Daltons having two moieties capable and where the ratio of more functional of reacting with the epoxy moieties of the di-epoxy resin and calculated on a molar basis of 12) New claim 2 from 3:1 to 12:1
  - -2) A coating-composition according to Claim 1-characterised in that the relative amount of mono-functional organic material to dicarboxylic acid, by weight, is from 3:1 to 12:1-
- 3) A coating composition according to Claim 1 or Claim 2 characterised in that the resin component of molecular weight less than 1000 Daltons comprises bis phenol A diglycidyl ether.
  - 4) A coating composition according to any one of the preceding Claims characterised in that the amount of bis phenol A diglycidyl ether extractable from a crosslinked coating of the coating composition is less than 0.3 micrograms/dm<sup>2</sup>
- 5) A coating composition according to any one of Claims 2 to 4 characterised in that the mono-functional organic material is a mono-carboxylic acid.
  - 6) A coating composition according to Claim 5 characterised in that the mono-carboxylic acid is tetradecanoic acid.
  - 7) A coating composition according to any one of the preceding Claims characterised in that the di-carboxylic acid is tartaric acid.

- 8) A coating composition according to any one of Claims 2 to 7 characterised in that the amount of reactive material comprises from 1 to 20% by weight of the modified epoxy resin.
- 9) A coating composition according to any one of Claims 2 to 8 characterised in that the5 modified epoxy resin has at least 30% of the number of epoxy groups as on the diepoxy resin from which it is derived.
- 10) A process for producing the modified epoxy resin as defined in any one of the preceding Claims comprising the steps of causing a diepoxy resin of epoxy equivalent weight of from 500 to 5000, formed by the reaction of bis phenol A diglycidyl ether and bis
  10 phenol A and containing minor amounts of resin components of molecular weight less than 1000 Daltons to react with a mono-functional organic material of molecular weight at least 100 Daltons and a dicarboxylic acid of molecular weight less than 300 Daltons.
- 11) A process according to Claim 10 characterised in that the mono-functional organic material is reacted with the diepoxy resin in a first step, the resulting product being reacted with the dicarboxylic acid in a later step.
  - 12) A metal container coated with the coating composition according to any one of Claims 1 to 9.
- 13) A process of producing a crosslinked coating on a metal container characterised in that it comprises the steps of applying a coating according to any one of Claims 1 to 9 and 20 causing the coating to crosslink.
  - 14) A modified epoxy resin as defined in any one of Claims 1 to 9.
  - 15) The use of a modified epoxy resin for reducing the amount of bis phenol A diglycidyl ether extractable from a crosslinked coating composition on the interior surface of a metal container to less than 0.3 micrograms/dm<sup>2</sup>, said modified epoxy resin being as defined in

25 Claim 14

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ii) from 0.1 to 20 parts of reactive material

characterised in that

a) the di-epoxy resin contains minor amounts of resin components of molecular weight
 less than 1000 Daltons, including bis phenol A diglycidyl ether

and the reactive material comprises

- b) mono-functional organic material of molecular weight at least 100 Daltons having one moiety capable of reacting with the epoxy moieties of the di-epoxy resin and
- capable of reacting with the epoxy moieties of the di-epoxy resin and where the ratio of mono-functional organic material to dicar boxylic acid calculated on a molar basis is from 3:1 to 12:1.

  In an alternative solution to the problem described above there is provided (New By minor amount is meant that resin components of molecular weight less than 1000 Daltons, including BADGE comprise less than 50% by weight of the total di-epoxy resin solids, preferably less than 20%, more preferably less than 2%, even more preferably from 0.005% to 1%, still more preferably from 0.01% to 0.5% and most preferably from 0.03% to 0.3%.

Preferably the resin component comprises BADGE.

More preferably the reactive moieties of the organic material react with the epoxy moieties of the di-epoxy resin, especially with the epoxy moieties of the minor amounts of BADGE. This tends to increase the molecular weight of the resin thereby reducing the amounts of resin component below 1000 Daltons, including the extractable BADGE. Preferably the amount of resin components of molecular weight below 1000 Daltons extractable from the crosslinked film is below 125 micrograms/dm<sup>2</sup> of crosslinked coating, more preferably from 1 to 100 micrograms/dm<sup>2</sup> and most preferably from 1 to 65 micrograms/dm<sup>2</sup> as measured by the method below.

Preferably the amount of BADGE extractable from a crosslinked coating of the coating

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ii) from 0.1 to 20 parts of reactive material characterised in that

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- a) the di-epoxy resin contains minor amounts of resin components of molecular
   weight less than 1000 Daltons, including bis phenol A diglycidyl ether
   and the reactive material comprises
  - b) mono-functional organic material of molecular weight at least 100 Daltons having one moiety capable of reacting with the epoxy moieties of the di-epoxy resin and
- c) dicarboxylic acid of molecular weight less than 300 Daltons having two moieties
   capable of reacting with the epoxy moieties of the di-epoxy resin

In an alternative solution to the problem described above, there is a provided a protective crosslinkable coating composition comprising modified epoxy resin and crosslinker the modified epoxy resin being the reaction product, by weight, of

- 15 i) from 80 to 99.9 parts of di-epoxy resin of epoxy equivalent weight from 500 to 5000 and formed from the reaction of bis phenol A diglycidyl ether and bis phenol A and
  - ii) from 0.1 to 20 parts of reactive material characterised in that
- a) the di-epoxy resin contains minor amounts of resin components of molecular weight less than 1000 Daltons

and the reactive material comprises

- b) mono-functional organic material of molecular weight at least 100 Daltons having one moiety capable of reacting with the epoxy moieties of the di-epoxy resin and
- c) tartaric acid having two moieties capable of reacting with the epoxy moieties of the di-epoxy resin.

Preferably the resin component comprises BADGE.

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By minor amount is meant that resin components of molecular weight less than 1000 Daltons, including BADGE comprise less than 50% by weight of the total di-epoxy resin solids, preferably less than 20%, more preferably less than 2%, even more preferably from 0.005% to 1%, still more preferably from 0.01% to 0.5% and most preferably from 0.03% to 0.3% and where the ratio of mon-functional organic material to dicarboxylic acid calculated on a molar basis is from 3:1 to 12:1.

More preferably the reactive moieties of the organic material react with the epoxy moieties of the di-epoxy resin, especially with the epoxy moieties of the minor amounts of BADGE. This tends to increase the molecular weight of the resin thereby reducing the amounts of resin component below 1000 Daltons, including the extractable BADGE. Preferably the amount of resin components of molecular weight below 1000 Daltons extractable from the crosslinked film is below 125 micrograms/dm² of crosslinked coating, more preferably from 1 to 100 micrograms/dm² and most preferably from 1 to 65 micrograms/dm² as measured by the method below.

Preferably the amount of BADGE extractable from a crosslinked coating of the coating